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(54) **METHODE DE PRODUCTION D'OBJETS SOUS FORME DE DETERGENT, STABLES ET A DESAGREGATION
RAPIDE**

(54) **METHOD FOR THE PRODUCTION OF STABLE AND RAPIDLY DISINTEGRATING SHAPED DETERGENT
UNITS**

(57)

The present invention relates to a process for the production of shaped detergent and cleaning composition units through compression of a particulate detergent and cleaning composition that can be produced through mixing of a detergent and cleaning composition granulate manufactured in one of the known ways with powdery upgrading components. Particularly stable shaped units that stand out for short disintegration times can be produced by mixing the granulate with the powdery upgrading components in a mixer and subjecting the mixture, after addition of the last component, to at least four mixer rotations during a sojourn time in the mixer of 1-300 seconds.



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Abstract

The present invention relates to a process for the production of shaped detergent and cleaning composition units through compression of a particulate detergent and cleaning composition that can be produced through mixing of a detergent and cleaning composition granulate manufactured in one of the known ways with powdery upgrading components. Particularly stable shaped units that stand out for short disintegration times can be produced by mixing the granulate with the powdery upgrading components in a mixer and subjecting the mixture, after addition of the last component, to at least four mixer rotations during a sojourn time in the mixer of 1-300 seconds.

METHOD FOR THE PRODUCTION OF STABLE AND RAPIDLY DISINTEGRATING SHAPED DETERGENT UNITS

The present invention relates to the production of shaped detergent and cleaning composition units. The invention relates, in particular, to a method for the production of detergent and cleaning-active shaped units that can be produced through compression shaping of particulate detergent and cleaning compositions and stand out for great solidity and, at the same time, favourable disintegration and dissolution characteristics.

Detergent and cleaning-active shaped units are produced by applying pressure to a compound to be compressed that is located in the hollow space of a press. In the simplest case of shaped unit production - hereinafter, for reasons of simplicity, referred to as tablet pressing - the compound to be shaped into tablets is directly compressed, i.e. without previous granulation. The advantages of this so-called direct tablet pressing consist in its simple and cost-efficient application resulting from the fact that no further process steps and, hence, no further machines are required. These advantages, however, are accompanied by disadvantages. A powdery compound to be subjected to direct tablet pressing must, for instance, be characterised by sufficient plastic ductility and good flow properties; additionally, absolute absence of demixing tendencies during storage, transport and charging of the mould is required. These three preconditions are extremely difficult to control in many substance compounds so that direct tablet pressing is, especially in the context of the production of detergent and cleaning composition tablets, applied very rarely.

The common method to produce detergent and cleaning composition tablets uses powdery components ("primary particles") as a starting material which is then agglomerated or granulated, respectively, into secondary particles with a greater particle diameter by application of appropriate methods. These granulates or mixtures of different granulates are then mixed with individual additive substances and sub-

jected to tablet pressing. In this respect, the characteristics of the granulates are of decisive importance for the physical characteristics of the shaped units: particle size, content of moisture and other parameters that can be controlled in the granulates contribute decisively to the characteristics of the resulting shaped units. In this respect, two physical characteristics of shaped units are of decisive importance in connection with shaped detergent and cleaning composition units: hardness and speed of disintegration. In the course of tablet production, application of correspondingly high pressure allows the production of shaped units of any desired stability, but the time required for disintegration of the resulting shaped unit rapidly grows with the pressure applied. As a consequence of the fact that the desired characteristics of a hard tablet characterised by stability during transport and handling that will nevertheless disintegrate rapidly are opposed to each other, the production of detergent and cleaning composition tablets generally is faced with the problem of overcoming, to the greatest possible extent, the dichotomy between hardness and disintegration.

As far as the production of granulates is concerned, prior art comprises an almost endless amount of written material ranging from patent documents to complete monographs of granulating technology.

EP-B-642 576 (Henkel) discloses a method for the continuous production of granulates wherein the product flows horizontally through a first, low-speed mixer/granulator (circumferential speed of the mixing tools 2 – 7 m/s) where it is pre-granulated and then vertically through a second, high-speed mixer/granulator where it is fully granulated (circumferential speed of the mixing tools > 8 m/s).

The combination of slow and fast mixers with different sojourn times of the products in the mixing granulators is also extensively described in prior art. The European patent application **EP-A-264 049** (BAYER AG), for example, describes a method for the production of granulates where the powder to be granulated is granulated, add-

ing a granulating liquid, first in a fast and then in a slow mixing granulator and subsequently dried in a fluidised bed. The product stays in the fast mixer (speed 800 – 3000 r.p.m.) for a period of 0.5 to 60 seconds and in the slow mixer (speed 60 to 250 r.p.m.) for another period of 60 to 300 seconds.

The adaptation of the above-mentioned procedure for the production of detergent granulates is described in EP-A-367 339 (Unilever). This document discloses the production of detergent granulates with bulk densities exceeding 650 g/l through treatment of a powdery starting material in a high-speed mixer (speed 100-2500 r.p.m., sojourn time 5-30 s), subsequent mixing in a slow mixer (speed 40-160 r.p.m., sojourn time 60-600 s) and final drying.

EP-A-390 251 (Unilever) expands the latter method by adding 0.1 to 40 percent in weight of a powder between the fast and the slow mixer. This measure is intended to minimise formation of particles with excessively large particle diameters.

Shaped detergent and cleaning composition units are produced by compressing particulate detergent and cleaning compositions that consist, at least in part, of granulates. Shaped detergent and cleaning composition units as well as methods for their production are also extensively described in prior art. EP-A-0 522 766 (Unilever), for example, discloses shaped units consisting of a compacted, particulate detergent composition that contains tensides, builders and substances enhancing disintegration (e.g. cellulose-based), wherein at least part of the particles is coated with the disintegration enhancing substance which, during dissolution of the shaped units in water, displays both binding and disintegrating effects. The same document points out the general problem of producing shaped units of adequate stability that are, at the same time, easily soluble. The particle-size of the compound to be compressed should exceed 200 µm, and the upper and the lower limits for the individual particle sizes should not be more than 700 µm apart. The shaped units are produced by

mixing a detergent and cleaning composition granulate produced in one of the known ways with powdery upgrading agents and subsequent compression shaping.

Additional documents relating to the production of shaped detergent units are EP-A-0 716 144 (Unilever), which describes shaped units with an external coating of water-soluble material, and EP-A-0 711 827 (Unilever), which specifies a citrate of defined solubility as an ingredient.

The application of binding agents which exert a disintegrating effect, if any, (especially polyethylene glycol) is disclosed in EP-A-0 711 828 (Unilever), which describes shaped detergent units that are produced by compressing a particulate detergent composition at temperatures between 28°C and the melting point of the binding material, the pressing always taking place below the melting temperature. Examples mentioned in this document indicate that shaped units manufactured according to the disclosed method display a higher resistance to breaking where the pressing process was performed at a higher temperature.

Detergent tablets where individual ingredients are arranged in discrete regions separate from the rest are also described in EP-A-0 481 793 (Unilever). The detergent tablets disclosed in this document contain sodium percarbonate arranged in a discrete region separate from all other components that might exert an influence on its stability.

The production methods for detergent and cleaning-active shaped units indicated in prior art consist of compressing shaping the respective substances, which partly takes place at different temperatures. As further influencing parameters, prior art only mentions physical characteristics of the compound compressed such as the particle size, the spatial distribution of the individual components of physical characteristics of individual components.

Prior art, however, does not describe how purposeful preparation of the particulate detergent and cleaning composition to be compressed can exert a positive influence on the physical characteristics of the resulting shaped detergent and cleaning composition units.

The objective of the present invention consists in providing, in addition to the selection of individual components, another influencing factor that can be used to improve the physical characteristics of shaped detergent and cleaning composition units. The present invention, in particular, focussed on the task of providing a method to supply, through purposeful preliminary treatment of the compound to be compressed, detergent and cleaning composition tablets that are hard and yet disintegrate quickly.

It has been found that compliance with certain mixing times is essential, when mixing the granular components with the powdery mixing components in the course of the production of the particulate detergent and cleaning composition to be compressed, in order to produce tablets characterised by great hardness and favourable disintegration characteristics at the same time.

The object of the present invention is, therefore, a method for the production of shaped detergent and cleaning composition units by mixing a detergent and cleaning composition granulate manufactured in one of the known ways with powdery upgrading components and subsequent compression shaping, wherein the mixing of the granulate with the powdery upgrading components takes place in a mixer and the mixture is, after addition of the final component and for a sojourn time between one and 300 seconds, subjected to at least four mixer revolutions.

The detergent and cleaning composition granulate used in the context of the process disclosed hereunder can be manufactured in different ways and can contain varying amounts of the common ingredients of detergents and cleaning agents. The granu-

lar particles can be differently shaped depending on the production method, nearly spherical granulates often being referred to as pellets.

The range of possible granulation methods comprises, for example, wet granulation, dry granulation or granulation on the basis of melt cooling, of which wet granulation represents the most common granulation method as it is subject to fewest limitations and with the greatest probability yields granulates with favourable properties. In the context of this granulation method, we distinguish between granulation involving the use of glue and granulation involving crust formation, and between granulation by building up and granulation by breaking down of substances. In the first case, the distinction depends on whether the powdery compounds to be granulated are granulated with solutions of binding agents or glues on the one hand or with pure solvents or solvent mixtures on the other. In the second case, the distinction is made between processes where finer particles are combined into larger aggregates or larger units are comminuted into fine granulates.

Granulation can be performed with different devices such as fluid bed granulators, fast and slow mixing granulators, roller-type compactors, ring matrix presses, pellet presses and many others. Extrusion, which can be used in the form of both wet and dry granulation, can also be applied for the production of the granulates used in the context of the method disclosed hereunder.

The granulates can be subjected to the known types of aftertreatment. A drying step may, in particular, be required after wet granulation, or the granular particles can be treated with a view to improving their surface characteristics, e.g. by applying a layer of a finely pulverised substance to prevent granular particles from sticking together.

The granulates can consist of all ingredients commonly contained in detergents and cleaning compositions. This includes, in particular, builders and tensides, but also bleaching agents and bleach activators, enzymes, co-builders, foam inhibitors, opti-

cal brightening agents, phosphonates, polymers as well as colorants and scents. In the framework of the invention disclosed hereunder, a process is preferred where detergent and cleaning composition granulates manufactured in one of the known ways contain tenside(s), builder(s) as well as optional other ingredients commonly used in detergents and cleaning compositions.

The expert will encounter no difficulty at all in including individual detergent and cleaning composition ingredients in the course of granulate production or during mixing of the granulates with powdery upgrading components. Depending on the desired characteristics of the shaped units, it is therefore possible to integrate the detergent and cleaning composition ingredients into the shaped unit either via the granulate or via the powdery upgrading components. In this connection, it is preferred to integrate detergent and cleaning composition ingredients that could suffer deterioration during the granulation process into the shaped unit via the powdery upgrading components.

In a preferred process for the production of shaped detergent and cleaning composition units, one or several substances from the group of tensides, tenside compounds, builders, bleaching agents, bleach activators, enzymes, foam inhibitors, colorants and scents as well as binding agents and substances enhancing disintegration are added to the granulate in the form of a powdery upgrading component.

Significant variations are also possible as far as the quantities of granulate and powdery upgrading components are concerned. In the framework of the invention disclosed hereunder, a process is preferred where 30 - 80, or preferably 40 - 75, or optimally 50 - 70 percent in weight of granulate and 20 - 70, or preferably 25 - 60, or optimally 30 - 50 percent in weight of powdery components (all percentages relating to the quantity of the resulting mixture to be subjected to tablet pressing) are mixed with each other and the mixture is, after addition of the final component and during a

sojourn time of 1 -300, or preferably 10 - 180, or optimally 20 - 120 seconds, exposed to at least four mixer revolutions.

In order to make better use of existing granulation facilities or to use existing types of granulates, the detergent and cleaning composition granulate manufactured in one of the known ways can also be composed of two or more separately manufactured individual granulates. It is, for example, possible to combine a niotenside zeolite granulate with an anionic tenside silicate granulate and the powdery upgrading components without noting any reduction in quality whatsoever in comparison with one single granulate consisting of all four components. In the framework of the present invention, a process is preferred where the detergent and cleaning composition granulate manufactured in one of the known ways consists of two or several separately manufactured granulates.

In order to achieve an even distribution of granulate(s) and powdery upgrading components, the mixture to be subjected to tablet pressing must be subjected to at least four mixer revolutions. The time required for these revolutions is irrelevant. Both fast high-intensity mixers and slower mixers can be used. The sojourn time of the granulates must, irrespective of the type of mixer used, be less than 300 seconds. The upper time limit beyond which a negative impact on the characteristics of the tablets will occur is 300 seconds even though granulates are subjected to significantly more revolutions in a fast than in a slow mixer. For economic reasons, however, it can be desirable to adapt the sojourn times of the mixture to be subjected to tablet pressing in the mixer to the rotational speed of the device. When using slow mixers with speeds between 10 and 250 r.p.m., sojourn times of 1-300 seconds, or preferably 20 - 180 seconds, or optimally 30 - 150 seconds should be preferred. In the case of fast mixers with speeds between 250 and 3000 r.p.m., sojourn times of 1 - 300 seconds, or preferably 2 - 180 seconds, or optimally 3 - 90 seconds are recommended.

In order to achieve an even distribution of components, the mixture of granulate(s) and powdery components must be thoroughly blended. For this purpose, at least four mixer revolutions are required, though a higher number of revolutions can be useful from a process-engineering point of view. In the framework of the present application, the terms "revolve" and "revolutions" refer to the minimum number of required revolutions of the mixer shaft which, depending on the rotational speed of the device, creates more or less turbulence and carries along more or less of the product. At a mixer speed of 40 r.p.m., for instance, a minimum of four mixer revolutions means that the product to be compressed must stay in the mixer for a minimum period of 6 seconds.

In the framework of the present invention, a process is preferred wherein the mixture of granulate(s) and powdery components is subjected to at least four, or preferably at least eight, or optimally at least ten mixer revolutions.

The actual production of the shaped units subject of the present invention begins with dry mixing of the granulate and the powdery components, followed by shaping, in particular tablet pressing, which can be performed by applying conventional methods. For the production of the shaped units covered by the present invention, the preliminary mixture is compressed, resulting in a solid compressed unit, in a so-called female mould located between two dies. This process, hereinafter simply referred to as tablet pressing, consists of four stages: charging, compression (elastic deformation), plastic deformation and ejection.

The first step consists of charging the mould with the preliminary mixture, the quantity of product filled into the mould and, thus, the weight of the resulting shaped unit being determined by the position of the lower die and the shape of the pressing tool. Constant charging even at high throughput rates is preferably achieved by volumetric apportioning of the preliminary mixture. In the further course of tablet pressing, the upper die touches the preliminary mixture and continues to move in the direction

of the lower die. During this compression process, the distances between the individual particles of the preliminary mixture are reduced, the total volume of hollow space within the material between the two dies continuously decreasing. Plastic deformation, in the course of which the particles flow together and the shaped unit is created, occurs from a certain position of the upper die (and thus from a certain pressure exerted on the preliminary mixture) onwards. Depending on the physical characteristics of the preliminary mixture, part of the particles of the preliminary mixture is crushed and sintering of the preliminary mixture occurs as the pressure continues to increase. As the pressing speed increases, i.e. at high throughput rates, the elastic deformation phase is increasingly shortened, which means that the resulting shaped units can contain smaller or larger hollow spaces. In the course of the last step of tablet pressing, the finished shaped unit is pushed out of the mould by the lower die and removed by subsequent conveying equipment. At this moment, only the weight of the shaped unit is finally determined, as the pressed units can still change their shape and size as a consequence of physical processes (elastic relaxation, crystallographic effects, cooling, etc.).

Tablet pressing is performed in commercial tablets presses which can basically be equipped with single or double dies. In the latter case, pressure is exerted not only by the upper die, but the lower die also moves in the direction of the upper die during the compression process while the upper die exerts pressure from above. Eccentric tablets presses where the die(s) is/are mounted on an eccentric disc that is mounted on an axle with a certain rotational speed should be preferred for small production volumes. The movement of these press dies can be compared with the working method of a common four-stroke engine. Compression can be performed with one upper and one lower die each, but is also possible to have several dies mounted on one eccentric disk, the number of die recesses in this case being correspondingly increased. Depending on the type of machine, the throughput rates of eccentric presses vary between several hundred to a maximum of 3000 tablets per hour.

Rotary tablet presses where a larger number of moulds is arranged in a circle on a press plate are used for larger throughput numbers. Depending on the type of machine, the number of moulds varies between 6 and 55, larger moulds also being commercially available. An upper die and a lower die are allocated to each of the moulds on the press plate, active pressure being exerted by either the upper or the lower die alone or both dies at the same time. The press plate and the dies move around a common vertical axis, rail-like curved guides serving to move the dies into the positions for filling, compacting, plastic deformation and ejection in the course of the rotation. Additional holding-down pieces, lowering rails and lifting guides are used to support said curved guides in places where particularly significant lifting or lowering of the dies is required (filling, compacting, ejection). Filling of the moulds is performed by means of an immovable charging device, the so-called filling shoe, which is connected with a storage tank containing the preliminary mixture. The pressure exerted on the preliminary mixture can be individually adjusted by modifying the stroke lengths of the upper and lower dies, pressure being built up as the die shaft heads pass adjustable pressure rollers.

Rotary presses can be equipped with two filling shoes in order to increase their throughput rates, which means that manufacturing of one tablet requires the mould to perform only half a rotation. For the production of shaped units consisting of two or more layers, several filling shoes are arranged one after the other in such a way that the first layer, being only slightly compacted, is not ejected prior to further filling. In this way, tablets with an outer enclosure or spherical insert that consist of several concentric layers can be produced by selecting a corresponding process design, the upper side of the core or core-layers remaining uncovered and, thus, visible in the case of tablets with a spherical insert. Rotary tablet presses can also be equipped with single or multiple tools so that it is, for example, possible to simultaneously compact material contained in an external circle of 50 and an internal circle of 35

recesses. The throughput rates of modern rotary tablet presses amount to more than one million of shaped units per hour.

Tablet pressing machines suitable for application in the framework of the present invention are, for example, offered by the following companies: Firmen Apparatebau Holzwarth GbR, Asperg, Wilhelm Fette GmbH, Schwarzenbek, Hofer GmbH, Weil, KILIAN, Cologne, KOMAGE, Kell am See, KORSCH Pressen GmbH, Berlin, Mapag Maschinenbau AG, Bern (CH) and Courtoy N.V., Halle (BE/LU). A particularly suitable machine is, for example, the hydraulic double press "Hydraulische Doppel-druckpresse HPF 630" offered by the company LAEIS, D.

The shaped units can be produced in a predetermined three-dimensional shape and a pre-determined size. The three-dimensional shape can be practically any shape with reasonable handling characteristics, which means that units can be shaped as slabs, sticks or bars, cubes, cuboids or similar three-dimensional structures with plane lateral surfaces or, in particular, in the form of cylinder-shaped elements with a circular or oval cross section. This latter design comprises all shapes ranging from tablets to compact cylinders wherein the ratio that the height bears to the diameter exceeds 1, particularly homogeneous distribution of density in the shaped units being achieved where the ratio that the diameter bears to the height is approximately 4.

The apportioned pressed units can be designed as separate individual elements of which each contains the predetermined dose of the detergent and/or cleaning composition. However, it is also possible to produce pressed units that contain several times said dose, in which case the units should, in particular, be provided with pre-determined breaking lines allowing splitting of the unit into smaller units each containing a predetermined dose of the product. For application of laundry detergents in washing machines of the type commonly used in Europe, i.e. washing machines equipped with a horizontally arranged mechanism, shaping of the apportioned

pressed units in the form of tablets, cylinders or cuboids can be recommendable for functional reasons, the diameter/height ratio preferably being in the range of approx. 0.5 : 2 to 2 : 0.5. Usual commercial hydraulic presses, eccentric presses or rotary presses are appropriate devices, in particular for the production of such pressed units.

The three-dimensional shape of a different type of shaped units is adapted, in terms of its dimensions, to the soap dispenser of usual commercial household washing machines, thus allowing direct placing of the shaped units, without a dosing device, in the soap dispenser where they dissolve as water flows through the dispenser into the machine. It is, of course, also easily possible to use shaped detergent units in combination with a dosing device.

Another preferred type of shaped unit that can be manufactured is characterised by a slab or plate-like structure containing an alternating series of long thick and short thin segments, thus allowing individual segments to be broken off this "bar", at the predetermined breaking lines represented by the short thin segments, and put into the machine. The principle underlying the "bar-type" shaped detergent unit can also be translated into practice in the form of other geometric shapes such as vertically aligned triangles that are only longitudinally connected at one of their sides.

However, it is also possible to manufacture shaped units where the different components are not compacted into a homogeneous tablet but which are characterised by several layers, i.e. which comprise a minimum of two layers. In this connection, it is also possible to manufacture different layers that dissolve at different speeds. This can yield advantageous characteristics of the shaped units in terms of their practical application. If the shaped units, for example, contain components that negatively influence each other, it is possible to integrate one of the components concerned into a layer that dissolves faster and the other into another layer that dissolves more slowly so that the first component has already exerted its effect when the second

begins to dissolve. The layers of the shaped units can be stacked, which means that dissolution of the internal layer(s) begins at the sides of the shaped unit before complete dissolution of the outer layers. Alternatively, it is also possible to fully enclose the inner layer(s) by the respective outer layer(s), which prevents premature dissolution of components contained in the inner layer(s).

In another preferred version of the invention, the shaped unit consists of at least three layers, i.e. two outer and at least one inner layer, whereof at least one of the inner layers contains a peroxy bleaching agent while the top and bottom layer of the stack-type shaped unit and the outermost layers of the enclosure-type shaped unit are free from peroxy bleaching agent. Furthermore, it is also possible to spatially separate peroxy bleaching agents and bleach activators and/or enzymes, if any, within a shaped unit. Such multi-layered shaped units are characterised by the favourable feature that they can not only be applied via a soap dispenser or a dosing device put amidst the laundry. Instead, it is in such cases also possible to put the shaped unit into the washing machine in direct contact with the laundry without risking staining of fabrics by bleaching agents or similar damage.

Similar effects can also be achieved by coating individual components of the detergent and cleaning composition to be compressed or the entire shaped unit, respectively. For this purpose, the elements to be coated can, for example, be sprayed with aqueous solutions or emulsions or be subjected to melt film coating.

After compression, the shaped detergent and cleaning composition units are characterised by great stability. The resistance to breaking of cylindrical shaped units can be expressed by using the diametral fracture stress value. This value is calculated using the following formula:

$$\sigma = \frac{2P}{\pi D t}$$

In this formula, σ stands for the diametral fracture stress (DFS) indicated in Pa. P stands for the force N resulting in the pressure exerted on the shaped unit that causes breaking of the shaped unit. D is the diameter of the shaped units indicated in meters, and t the height of the shaped units.

In preferred variations of the process disclosed hereunder, the mixture of granulate(s) and powdery upgrading components to be compacted is subjected to compaction at a pressure of 10 - 150 N/cm², or preferably 15 - 100 N/cm², or optimally 20 - 100 N/cm², and a temperature of 10 - 80°C, or preferably 15 - 70°C, or optimally 20 - 60°C.

The following section contains a brief description of the most important ingredients of shaped detergent and cleaning composition units which can, in the context of the process disclosed hereunder, be contained in the granulates or in the powdery upgrading components. The granular components of the mixture to be compacted are manufactured in one of the familiar ways and one of the familiar compositions, the selection of ingredients always depending on the desired purpose of the shaped units.

For the shaped detergent and cleaning composition units disclosed hereunder, it is possible to use anionic, non-ionic, cationic and/or amphoteric tensides. From an application-related point of view, mixtures of anionic and non-ionic tensides where the share of anionic tensides should exceed the share of non-ionic tensides are preferable. The total tenside content of the shaped units amounts to 5 - 60 percent in weight in relation to the weight of the shaped unit, tenside contents exceeding 15 percent in weight being preferred.

Examples for possible anionic tensides are sulphonate-type or the sulphate-type tensides. Preferable sulphonate-type tensides are C₉₋₁₃-alkylbenzene sulphonate, olefin sulphonates, i.e. mixtures of alkene sulphonates and hydroxyalkene sulpho-

nates as well as disulphonates of the type produced from C_{12-18} -monoolefins with a terminal or internal double bond by sulphonation with gaseous sulphur trioxide and subsequent alkaline or acid hydrolysis of the sulphonation products. Further suitable substances are alkane sulphonates produced from C_{12-18} -alkanes, for examples, through sulphochlorination or sulfoxidation with subsequent hydrolysis or neutralisation, respectively. Esters of α -sulphonic fatty acids (ester sulphonates) such as α -sulphonated methyl esters of hydrated coconut, palm kernel or tallow fatty acids.

Further suitable anionic tensides are represented by sulphurised fatty acid glycerine esters. Fatty acid glycerine esters are monoesters, diesters and triesters as well as compounds thereof of the type produced through esterification of one monoglycerine with 1 to 3 mol of fatty acid or transesterification of triglycerides with 0.3 to 2 mol of glycerine. Preferred sulphurised fatty acid glycerine esters are the sulphurisation products of saturated fatty acids with 6 – 22 carbon atoms such as caproic acid, caprylic acid, capric acid, myristic acid, lauric acid, palmitic acid, stearic acid or behenic acid.

Among the alk(en)yl sulphates, preference is given to alkali and, in particular, sodium salts of sulphuric acid semi-esters of C_{12} - C_{18} -fatty alcohols, e.g. of coconut fatty alcohol, tallow fatty alcohol, lauryl alcohol, myristyl alcohol, cetyl alcohol or stearyl alcohol, or of C_{10} - C_{20} -oxo-alcohols and semi-esters of secondary alcohols of the same chain lengths. Additional preferred substances are alk(en)yl sulphates of the chain lengths indicated above which contain a synthetic straight-chain alkyl rest manufactured on a petrochemical basis and are characterised by similar degradation behaviour as the adequate compounds on the basis of fat-chemical raw materials. From the point of view of washing, preference is given to C_{12} - C_{16} -alkyl sulphates and C_{12} - C_{15} -alkyl sulphates as well as C_{14} - C_{15} -alkyl sulphates. 2,3-alkyl sulphates that can be manufactured, for example, according to the US patents 3,234,258 or 5,075,041 and are supplied by the Shell Oil Company under the name DAN® also represent suitable anionic tensides.

Sulphuric acid monoesters of straight-chained or branched-chain C_{7-21} -alcohols ethoxylated with 1 – 6 mol of ethylene oxide such as 2-methyl-branched C_{9-11} -alcohols with an average of 3.5 mol of ethylene oxide (EO) or C_{12-18} -fatty alcohols with 1 – 4 EO are suitable. Because they give rise to significant foam development, they are only used in relatively small amounts in cleaning compositions, e.g. in quantities of 1 – 5 percent in weight.

Further suitable anionic tensides are represented by the salts of alkyl sulphonic succinic acid which are also called sulphonic succinates or sulphonic succinic acid esters and are monoesters and/or diesters of sulphonic succinic acid with alcohols, or preferably fatty alcohols, or optimally, ethoxylated fatty alcohols. The preferred sulphonic succinates contain a C_{8-18} -fatty alcohol rests or mixtures of the latter. The particularly preferred sulphonic succinates contain a fatty alcohol rest derived from ethoxylated fatty alcohols which by themselves represent non-ionic tensides (see description below). In this connection, particular preference is given to sulphonic succinates with fatty alcohol rests derived from ethoxylated fatty alcohols with a narrowed distribution of homologues. It is also possible to use alk(en)yl succinic acid with preferably 8 – 18 carbon atoms in the alk(en)yl chain or its salts.

Further suitable anionic tensides are, in particular, soaps. Suitable soaps are saturated fatty acid soaps such as the salts of lauric acid, myristic acid, palmitic acid and stearic acid, hydrated erucic acid and behenic acid as well as mixtures of different soaps derived from natural fatty acids, e.g. coconut, palm kernel or tallow fatty acids.

Anionic tensides including soaps can be used in the form of their sodium, potassium or ammonium salts as well as in the form of soluble salts of organic bases such as mono-, di- or triethanolamine. Anionic tensides should preferably be used in the form of their sodium or potassium salts or, optimally, in the form of their sodium salts.

Where preliminary mixtures containing fatty alcohol sulphates as anionic tensides are manufactured in the framework of the process disclosed hereunder, fatty alcohol sulphates should preferably be integrated into the preliminary mixture to be compressed via the powdery upgrading components. Preference is, in this respect, given to the use of fatty alcohol sulphate compounds with an active substance content of at least 30 percent in weight. The process disclosed hereunder yields particularly favourable shaped units when the powdery component containing the fatty alcohol sulphate is added to the product in the mixer as the very last ingredient and when mixing times below 3 minutes are observed after addition of the fatty alcohol sulphate compound.

A preferred process is, therefore, one where the last powdery upgrading component added during the mixing process is a tenside compound containing at least 30 percent in weight (in relation to the tenside compound) of fatty alcohol sulphate and where the mixture is, after addition of the fatty alcohol sulphate compound, subjected to at least four mixer revolutions during a sojourn time in the mixer of 1 – 180 seconds, or preferable 10 – 150 seconds, or optimally 20 - 120 seconds.

Furthermore, it is also possible to integrate finely pulverised components into the process disclosed hereunder which stick to the surface of the granulate(s) and the other powdery upgrading components and serve to envelope these in a fine coat of powder. The advantage of adding this "coating powder" consists in the fact that it minimises or totally prevents the material to be compressed from sticking to the dies during the subsequent compression process. The substances suitable to be used as such finely pulverised coating powders include, for example, the builders described below, zeolites and, especially the zeolite X described in detail below being particularly suitable. In a further preferred process, a tenside compound containing at least 30 percent in weight (in relation to the tenside compound) of fatty alcohol sulphate as well as zeolite X are added as powdery upgrading components. In this case, zeolite X should preferably be added as the very last component, and the mixture

should, after addition of zeolite X, be subjected to at least four mixer rotations during a sojourn time in the mixer of 1 - 180 seconds, or preferably 10 - 150 seconds, or optimally 20 - 120 seconds.

The quantity of anionic tenside integrated into the shaped units via the preliminary mixture amounts, for example, to between 5 and 60 percent in weight. The anionic tensides should, however, preferably not be used alone but mixed with non-ionic tensides, the total content of anionic tensides in the shaped units in this case amounting to between 5 and 40 or, preferably, between 5 and 30 percent in weight.

In the framework of another preferred process, a fatty alcohol sulphate compound is added in the form of a powdery upgrading component in such a quantity that the compound to be compressed contains at least 2 percent in weight, or preferably at least 4 percent in weight, or optimally more than 5 percent in weight of fatty alcohol sulphate.

The non-ionic tensides preferably used are alkoxyated, or preferably ethoxylated, or optimally primary alcohols with preferably 8 to 18 carbon atoms and an average of 1 to 12 mol of ethylene oxide (EO) per mol of alcohol in which the alcohol rest can be linear or, preferably, methyl-branched in the 2-position or can contain methyl-branched rests in the mixture of the kind commonly contained in oxo-alcohol rests. Particular preference, however, is given to alcohol ethoxylates with linear rests from alcohols of native origin with 12 to 18 carbon atoms, e.g. from coconut, palm or tallow fatty alcohol or oleyl alcohol, and an average of 2 to 8 EO per mol of alcohol. The preferred ethoxylated alcohols comprise, for example, C₁₂₋₁₄-alcohols with 3 EO or 4 EO, C₉₋₁₁-alcohol with 7 EO, C₁₃₋₁₅-alcohols with 3 EO, 5 EO, 7 EO or 8 EO, C₁₂₋₁₈-alcohols with 3 EO, 5 EO or 7 EO and mixtures thereof, such as mixtures of C₁₂₋₁₄-alcohol with 3 EO and C₁₂₋₁₈-alcohol with 5 EO. The indicated ethoxylation levels represent statistical average values that can be a whole or a fractional number for a specific product. The preferred alcohol ethoxylates are characterised by a narrowed

distribution of homologues (narrow range ethoxylates, NRE). In addition to these non-ionic tensides, it is also possible to use fatty alcohols with more than 12 EO, for example tallow fatty alcohol with 14 EO, 25 EO, 30 EO or 40 EO.

Additionally, alkyl glycosides of the general formula $RO(G)_x$ can be used as further non-ionic tensides, R standing for a primary aliphatic rest with 8 to 22 or, preferably, 12 to 18 carbon atoms that is straight-chained or methyl-branched or, in particular, methyl-branched in the 2-position, while G is the symbol representing a glucose unit with 5 or 6 carbon atoms, preferably glucose. The level of oligomerisation x, which indicates the distribution of monoglycosides and oligoglycosides, is any desirable number between 1 and 10, preferably in the range between 1.2 and 4.

Another class of preferably used non-ionic tensides, which are applied either as the sole non-ionic tenside or in combination with other non-ionic tensides, consists of alkoxyated or, preferably, ethoxylated or ethoxylated and propoxylated fatty acid alkyl esters, preferably with 1 – 4 carbon atoms in the alkyl chain, in particular fatty acid methyl esters of the type described, for example, in the Japanese patent application JP 58/217598 or preferably manufactured applying the process described in the international patent application WO-A-90/13533.

Non-ionic tensides of the aminoxide type such as N-coconut-alkyl-N,N-dimethylaminoxide and N-tallow-alkyl-N,N-dihydroxyethylaminoxide, and of the fatty acid alkanolamide type can also be adequate. The quantity of these non-ionic tensides should preferably amount to not more than the quantity of ethoxylated fatty alcohols or, optimally, to not more than half thereof.

Additional suitable tensides are polyhydroxy fatty acid amides composed according to formula (I),



where RCO represents an aliphatic acyl rest with 6 to 22 carbon atoms, R^1 hydrogen, an alkyl or hydroxyalkyl rest with 1 to 4 carbon atoms and a [Z] a linear or branched polyhydroxyalkyl rest with 3 to 10 carbon atoms and 3 to 10 hydroxyl groups. Polyhydroxy fatty acid amides are known substances that can normally be produced through reductive amination of a reducing sugar with ammonia, an alkyl amine or an alkanolamine and subsequent acylation with a fatty acid, a fatty acid alkyl ester or a fatty acid chloride.

The group of the polyhydroxy fatty acid amides also comprises substances composed according to formula (II),



where R represents a linear or branched alkyl or alkenyl rest with 7 to 12 carbon atoms, R^1 a linear, branched or cyclical alkyl rest or an aryl rest with 2 to 8 carbon atoms and R^2 a linear, branched or cyclical alkyl rest or an aryl rest or an oxy-alkyl rest with 1 to 8 carbon atoms, preference being given to C_{1-4} -alkyl or phenyl rests, and [Z] stands for a linear polyhydroxy alkyl rest with an alkyl chain substituted with at least two hydroxyl groups or alkoxylated or, preferably, ethoxylated and propoxylated derivatives of said rest.

[Z] is preferably produced through reductive amination of a reducing sugar such as glucose, fructose, maltose, lactose, galactose, mannose or xylose. The N-alkoxy or

N-aryloxy-sustituted compounds can then, for example, be converted into the desired polyhydroxy fatty acid amides through reaction with fatty acid methyl esters in the presence of an alkoxide functioning as a catalyst according to the international application WO-A-95/07331.

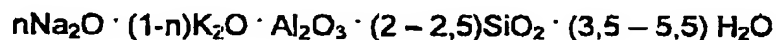
Among the substances eligible as builders, which can be contained in the shaped detergent and cleaning composition units covered by the invention, silicates, aluminium silicates (in particular zeolites), carbonates, salts or organic dicarbon and polycarbon acids as well as mixtures of the mentioned substances deserve particular mention.

Appropriate crystalline, stratified sodium silicates are defined by the general formula $\text{NaMSi}_x\text{O}_{2x+1}\text{H}_2\text{O}$, wherein M stands for sodium or hydrogen, x for a number between 1.9 and 4 and y for a number between 0 and 20, x values of 2, 3 or 4 being preferable. Crystalline stratified silicates of this type are, for example, described in the European patent application EP-A-0 164 514. Preference is given to crystalline stratified silicates of the indicated formula where M stands for sodium and x for a value of 2 or 3. Particular preference is given to both β - and δ -sodium disilicates $\text{Na}_2\text{Si}_2\text{O}_5\text{yH}_2\text{O}$, whereof β -sodium disilicate can, for example, be produced through application of the process described in the international patent application WO-A-91/08171.

It is also possible to use amorphous sodium silicates with a modul $\text{Na}_2\text{O} : \text{SiO}_2$ between 1:2 and 1:3.3, or preferably between 1:2 and 1:2.8, or optimally between 1:2 and 1:2.6, that are characterised by delayed dissolution and secondary washing properties. Delayed dissolution in comparison with conventional amorphous sodium silicates can be a result of different procedures such as surface treatment, compounding, compacting/compressing or drying. In the framework of the present invention, the term "amorphous" also signifies "X-ray-crystallographically amorphous". This means that silicates subjected to X-ray diffraction experiments do not yield

sharply defined X-ray reflexes of the type typical of crystalline substances but, if any, one or several maximums of the diffracted X-rays with a width of several degree units of the diffraction angle. However, it is definitely possible to achieve particularly good builder properties where silicate particles yield blurred or even sharply defined diffraction maximums in the context of X-ray diffraction experiments. This must be interpreted in the sense that the products contain micro-crystalline areas measuring between 10 and several hundred nm, preference being given to values of up to 50 nm and, in particular, of up to 20 nm. Such so-called "X-ray-crystallographically amorphous" silicates, which are also characterised by delayed dissolution in comparison with normal water glass, are, for example, described in the German patent application DE-A- 44 00 024. Preference is, in particular, given to compacted/compressed amorphous silicates, compounded amorphous silicates and dried X-ray-crystallographically amorphous silicates.

The fine-crystalline, synthetic zeolite containing bound water that is applied should preferably be zeolite A and/or P. The particularly preferred zeolite P is Zeolith MAP® (commercial product supplied by the company Crosfield). However, it is also possible to use zeolite X as well as mixtures of A, X and/or P. A co-crystallisate of zeolite X and zeolite A (containing approximately 80 percent in weight of zeolite X), which is distributed by the company CONDEA Augusta S.p.A under the trade name VEGOBOND AX® and defined by the formula below, is also commercially available and applicable in the framework of the process covered by the present invention:



Zeolite can be used as a spray-dried powder or in the form of an undried, stabilised suspension that still contains the humidity gained during the production process. Where zeolite is used in the form of the suspension, it is possible that the suspension contains small amounts of non-ionic tensides as stabilisers, e.g. between 1 and 3 percent in weight (in relation to the zeolite) of ethoxylated C₁₂-C₁₈-fatty alcohols

with 2 to 5 ethylene oxide groups, C₁₂-C₁₄-fatty alcohols with 4 to 5 ethylene oxide groups or ethyloxated isotridecyl alcohols. Appropriate zeolites have an average particle size below 10 μ m (volume distribution; measuring method: Coulter Counter) and contain preferably between 18 and 22, or optimally between 20 and 22 percent in weight of bound water.

Of course it is also possible to use the generally familiar phosphates as builders unless the application of this substance type should preferably be avoided for ecological reasons. Particularly appropriate are the sodium salts of orthophosphates, pyrophosphates and, in particular, tripolyphosphates.

Appropriate organic builders are, for example, polycarbon acids such citric acid, adipic acid, succinic acid, glutaric acid, tartaric acid, saccharic acids, amino carbon acids or nitrilotriacetic acid (NTA) that can be used in the form of their sodium salts unless such application is objected to for ecological reasons as well as mixtures thereof. Preferred salts are the salts of polycarbon acids such as citric acid, adipic acid, succinic acid, glutaric acid, tartaric acid, saccharic acids and mixtures thereof. These salts are used due to their builder characteristics and must not be considered as part of the gas development system as the salts are not able to release, for example, carbon dioxide out of hydrocarbonates.

Among the compounds serving as bleaching agents that yield H₂O₂ when dissolved in water, particular importance is attached to sodium perborate tetrahydrate and sodium perborate monohydrate. Additional substances that can be used as bleaching agents are, for example, sodium percarbonate, peroxyphosphate, citrate perhydrate as well as peracidic salts or peracids yielding H₂O₂ such as perbenzoate, peroxophthalate, diperazelaic acid, phthaliminoperic acid or diperdodecan diacid.

In order to achieve an improved bleaching effect at washing temperatures of 60 °C and below, it is possible to integrate bleach activators into the shaped detergent and

cleaning composition units. Substances that can be used as bleach activators are compounds that, in perhydrolytic conditions, yield peroxocarbon acids with preferably 1 to 10 carbon atoms or, in particular, 2 to 4 carbon atoms and/or substituted perbenzoic acid, if applicable. Suitable substances bear O and/or N-acyl groups with the number of carbon atoms indicated above and/or substituted benzoyl groups, if applicable. Preference is given to multiply acylated alkylene diamines such as, in particular, tetraacetyl ethylene diamine (TAED), acylated triazine derivatives such as, in particular, 1,5-diacetyl-2,4-dioxohexahydro-1,3,5-triazine (DADHT), acylated glykouriles such as, in particular, tetraacetylglykourile (TAGU), N-acylimides such as, in particular, N-nonanoylsuccinimide (NOSI), acylated phenolsulphonates such as, in particular, n-nonanoyl- or isononanoyloxybenzolsulphonate (n- or iso-NOBS, respectively), carbon acid anhydrides such as, in particular, phthalic acid anhydride, acylated polyhydric alcohols such as, in particular, triacetine, ethylenglykoldiacetate and 2,5-diacetoxy-2,5-dihydrofurane.

In addition to the conventional bleach activators or instead of such, it is also possible to integrate so-called bleach catalysts into the shaped units. These are bleach-enhancing transitional metal salts or transitional metal complexes, respectively, such as Mn, Fe, Co, Ru or Mo salene complexes or carbonyl complexes. Mn, Fe, Co, Ru, Mo, Ti, V and Cu complexes with nitrogen-containing tripod ligands as well as a Co, Fe, Cu and Ru amine complexes can also be used as bleach catalysts.

Possible foam inhibitors, which can be part of component b) or altogether make up component b), are, for example, soaps of natural or synthetic origin with a high content of C₁₈₋₂₄-fatty acids. Appropriate non-tensidic foam inhibitors are, for example, organopolysiloxanes and mixtures thereof with microfine, possibly silanated silicic acid or bistearylethylene diamide. Advantages are also offered by mixtures of different foam inhibitors, e.g. mixtures of silicones, paraffines or waxes. Foam inhibitors should preferably be bound to a granular, water-soluble or in water dispersible car-

rier substance. Preference in this respect is given to mixtures of paraffines and bistearylethylene diamides.

In addition, shaped detergent and cleaning composition units can also contain components that facilitate the removal of oil and grease from textile materials (so-called soil repellents). This effect becomes especially apparent when a textile material previously washed several times with a detergent of the type covered by the present invention that contains this oil and grease solving component is soiled. The preferred oil and grease-solving components comprise, for example, non-ionic cellulose ethers such as methyl cellulose methylhydroxy-propylcellulose with a share of methoxyl groups amounting to 15 to 30 percent in weight and a share of hydroxypropoxyl groups amounting to 1 to 15 percent in weight (both in relation to the non-ionic cellulose ether) as well as the polymers of phthalic acid and/or terephthalic acid or derivatives thereof described in prior art, in particular polymers of ethylene terephthalates and/or polyethylene glycolterephthalates or anionic and/or non-ionic modified derivatives thereof. Particular preference in this respect is given to sulphonated derivatives of phthalic acid and terephthalic acid polymers.

The range of enzymes that can be used contains enzymes from the class of proteases, lipases, amylases, cellulases or mixtures thereof. Enzymatic agents produced from bacteria strains or fungi such as *Bacillus subtilis*, *Bacillus licheniformis* und *Streptomyces griseus* are particularly suited. Preference is given to proteases of the subtilisine-type and, in particular, to proteases produced from *Bacillus lentus*. Of particular interest in this respect are mixtures of enzymes such as mixtures of protease and amylase or protease and lipase or protease and cellulase or cellulase and lipase or protease, amylase and lipase or protease, lipase and cellulase, but in particular cellulose-containing mixtures. Peroxidases and oxidases also proved appropriate in several cases. The enzymes can be adsorbed onto carrier substances and/or be embedded in enclosing substances in order to protect them against premature disintegration. The share of enzymes, mixtures of enzymes or enzyme

granulates in the shaped units produced according to the present invention can, for example, amount to about 0.1 to 5 or, preferably, to 0.1 to about 2 percent in weight.

As optical brightening agents, it is possible to integrate into the shaped units derivatives of diaminostilbendisulphonic acid or the alkali metal salts thereof. Suitable substances in this respect are, for example, salts of 4,4'-bis(2-anilino-4-morpholino-1,3,5-triazinyl-6-amino)stilben-2,2'-disulphonic acid or similarly composed compounds which bear a diethanol amino group, a methyl amino group, an anilino group or a 2-methoxy ethylamino group instead of the morpholino group. Additionally, brightening agents from the substituted diphenyl styryle type can be present such as, for example, the alkali salts of 4,4'-bis(2-sulphostyryl)-diphenyl, 4,4'-bis(4-chlor-3-sulphostyryl)-diphenyl, or 4-(4-chlorine styryl)-4'-(2-sulphostyryl)-diphenyl. Furthermore, it is also possible to use mixtures of the above-mentioned brightening agents..

Colorants and scents are added to the products covered by the present invention in order to improve the aesthetic impression created by the products and to provide the buyer with a product that performs not only a washing and cleaning function but is also "typical and unmistakable" in terms of its optical and sensory characteristics. The perfume oils or scents, respectively, can consist of various compounds of odoriferous substances such as, for example, synthetic products of the ester, ether, aldehyde, ketone, alcohol or hydrocarbon type. Ester-type compounds of odoriferous substances are, for example benzyl acetate, phenoxyethyl isobutyrate, p-tert-butylcyclohexyl acetate, linalyl acetate, dimethylbenzylcarbinyl acetate, phenylethyl acetate, linalyl benzoate, benzyl formate, ethylmethylphenyl glycinate, allylcyclohexyl propionate, styrallyl propionate and benzyl salicylate. The ether group comprises, for example, benzylethyl ether and the aldehyd group linear alkanals with 8 to 18 carbon atoms citral, citronellal, citronellyloxyacetaldehyde, cyclamenaldehyde, hydroxycitronellal, lillal and bourgeonal. The ketone group comprises, for example, jonones, α -isomethyl ionone and methylcedryl ketone, the alcohol group anethol, citronellol, eugenol, geraniol, linalool, phenylethyl alcohol and terpineol, and the hy-

drocarbon group mainly the terpenes such as limonen and pinen. Preference, however, is given to mixtures of different odoriferous substances which together result in an attractive scent. Such perfume oils can, of course, contain mixtures of odoriferous substances of the type produced from plants such as, for example, pine, citrus, jasmine, patchouli, rose or ylang-ylang oil. Further possible substances are muscateller oil, salvia oil, camomile oil, cloves oil, melissa oil, mint oil, cinnamon leaf oil, tilia oil, juniper berry oil, vetiver oil, olibanum oil, galbanum oil and labdanum oil as well as orange blossom oil, neroli oil, orange peel oil and sandalwood oil.

The content of colorants contained in the shaped units covered by the present invention is normally 0.01 in weight while scents can amount to up to 2 percent in weight of the entire formula.

Example:

In order to produce detergent tablets, a detergent granulate (composition as indicated in table 1) was filled into different types of mixers, sprayed with perfume and subsequently mixed with the upgrading components indicated in table 2. Prior to tablet pressing, 4 percent in weight of cellulose (disintegration enhancing agent) and 1 percent in weight of zeolite (coating powder component) were mixed with the preparation (all percentages indicated in relation to the weight of the resulting shaped unit). The compound was mixed for respective total mixing times of 30 seconds and 3 minutes in each of three different mixer types. The results summarised in table 3 show that detergent tablets manufactured from the preliminary mixture that was mixed longer took significantly longer to disintegrate in spite of much "softer" compression.

Table 1: Composition of the tenside granulate [percent in weight]:

C ₉₋₁₃ alkylbenzol sulphonate	22.0
C ₁₂₋₁₈ -fatty alcohol with 7 EO	6.2
Soap	1.6
Zeolite A	31.3
Sodium carbonate	18.8
Sodium silicate	5.5
Acrylic acid - maleic acid copolymer	5.5
Optical brightening agent	0.3
Salts / water	Rest

Table 2: Composition of the shaped detergent and cleaning composition units [percent in weight]:

Tenside granulate	65.2
Perfume	0.5
Sodium perborate	16.0
Tetraacetylene diamine (TAED)	7.3
Foam inhibitor	3.5
Enzymes	2.5
Cellulose	4.0
Zeolite A or zeolite X	1.0
Salts/water	Rest

The hardness of the tablets was measured through deformation of the tablet until breaking, the force acting on the lateral surfaces of the tablet as well as the maximum force sustained by the tablet being determined.

For the determination of tablet disintegration, the tablet was placed in a beaker glass of water (600 ml water, temperature 30°C) and the time until complete disintegration of the tablet without mechanic intervention was measured.

Table 3 contains a summary of the corresponding experimental data:

Table 3: Detergent tablets [physical specifications]

Tablet	Example 1	Example 1	Example 2	Example 2	Example 3	Example 3
Mixing time	30 sec	6 min	30 sec	6 min	30 sec	6 min
Tablet hardness	30 N	20 N	30 N	20 N	30 N	20 N
Tablet disintegration	< 30 sec	> 60 sec	< 30 sec	> 60 sec	< 30 sec	> 60 sec

Example 1: Lödige FM 130 D, Gebrüder Lödige Maschinenbau, D (blade mixer)

Example 2: Bolz Summiz ML 003, Fa. Helpman Verfahrenstechnik, Wangen, D
(conical spiral mixer)

Example 3: Pegasus PG 120, Fa. Dinnissen, Sevenum, NL (paddle mixer)

In the course of several further series of experiments, detergent tablets containing fatty alcohol sulphate were produced. fatty alcohol sulphate (FAS) in some of the experiments being added at the very end in the form of a powdery upgrading component while zeolite X was in other cases added to the compound in the mixer as a coating powder after addition of powdery FAS. In another experiment, FAS was added in the form of a granulate.

Table 4: Composition of the shaped detergent and cleaning composition units (example 4) [percent in weight]:

Tenside granulate	60.5
Perfume	0.5
Sodium perborate	16.0
Tetraacetylene diamine (TAED)	7.3
Foam inhibitor	3.5
Enzymes	2.5
Cellulose	4.0
Zeolite A or zeolite X	1.0
C ₁₂₋₁₈ -fatty alcohol sulphate, 96%*	4.7
Salts / water	Rest

*: Fatty alcohol sulphate compound with 96% active substance, 2% sodium carbonate and 2% water

In this example, the fatty alcohol sulphate compound was added as the last component and the product was mixed in a Lödige FM 130 D mixer. The mixing times after addition of the FAS were measured, and the physical specifications of the shaped detergent and cleaning composition units after compression were determined. The experimental data is summarised in table 6.

In a further example, 1 percent in weight of zeolite X was added as a coating powder after addition of 3.8 percent in weight of the FAS compound used in example 4, and the mixing was performed in a Lödige FM 130 D mixer. The mixing times after

addition of zeolite X were measured, and the physical specifications of the shaped detergent and cleaning composition units after compression were determined. The experimental data is summarised in table 6.

FAS can, in the framework of the present invention, also be used in the form of a tenside granulate. For this purpose, the tenside granulate used in example 6 was modified as follows. Instead of 6.2 percent in weight of C₁₂₋₁₈-fatty alcohol with 7 EO, a quantity of 3.2 percent in weight of 96% FAS compound and 3.0 percent in weight of C₁₂₋₁₈-fatty alcohol with 7 EO were used. This granulate was mixed with the other components in a Lödige FM 130 D mixer, the mixing time after addition of zeolite X (last component, coating powder) being measured. The composition of the shaped detergent and cleaning composition units is indicated in table 5, the physical specifications of the shaped detergent and cleaning composition units of examples 4, 5 and 6 are summarised in table 6.

Table 5: Composition of the shaped detergent and cleaning composition units (example 6) [percent in weight]:

Tenside granulate*	64.5
Perfume	0.45
Sodium perborate	16.0
Tetraacetylene diamine (TAED)	7.3
Foam inhibitor	3.5
Enzymes	2.5
Cellulose	4.0
Zeolite X	2.0
Salts / water	Rest

*: As indicated in table 1 containing 3.2 percent in weight of 96% FAS compound and 3.0 percent in weight of C₁₂₋₁₈-fatty alcohol with 7 EO instead of 6.2 percent in weight of C₁₂₋₁₈-fatty alcohol with 7 EO.

Table 6: Detergent tablets [physical specifications]

Tablet	Example 4	Example 4	Example 5	Example 5	Example 6	Example 6
Mixing time	5 sec	360 sec	120 sec	450 sec	180 sec	480 sec
Tablet hardness	33 N	31 N	34 N	33 N	35 N	34 N
Tablet disintegration	25 sec	> 60 sec	15 sec	> 60 sec	20 sec	> 60 sec

Claims:

1. Process for the production of shaped detergent and cleaning composition units through mixing of a detergent and cleaning composition granulate manufactured in one of the known ways with powdery upgrading components and subsequent compression shaping, characterised in that mixing of the granulate with the powdery upgrading components takes place in a mixer and the mixture is, after addition of the last compound, subjected to at least four mixer rotations during a sojourn time in the mixer of 1 – 300 seconds.
2. Process according to Claim 1, characterised in that the detergent and cleaning composition granulate manufactured in one of the known ways contains tenside(s), builder(s) as well as optional additional ingredients commonly contained in detergent and cleaning compositions.
3. Process according to one of Claims 1 or 2, characterised in that one or several substances from the group of tensides, tenside compounds, builders, bleaching agents, bleach activators, enzymes, foam inhibitors, colorants and scents as well as binding agents and disintegration enhancing agents are added as powdery upgrading components during the mixing process.
4. Process according to one of Claims 1 to 3, characterised in that 30 – 80 percent in weight, or preferably 40 – 75 percent in weight, or optimally 50 – 70 percent in weight of granulate and 20 – 70 percent in weight, or preferably 25 – 60 percent in weight, or optimally 30 – 50 percent in weight of powdery components (all percentages indicated in relation to the quantity of the resulting mixture to be compressed) are mixed with each other in the course of the mixing process and that the mixture is, after addition of the last component, subjected to at least four mixer rotations during a sojourn time in the mixer of

- 1 - 300 seconds, or preferably 10 - 180 seconds, or optimally 20 - 120 seconds.
5. Process according to one of Claims 1 to 4, characterised in that the detergent and cleaning composition granulate manufactured in one of the known ways consists of two or more separately produced individual granulates.
 6. Process according to one of Claims 1 to 5, characterised in that the mixing of the granulate with the powdery components takes place in a slowly moving mixer at a speed between 10 and 250 r.p.m. and during a sojourn time in the mixer of 1 - 300 seconds, or preferably 20 - 180 seconds, or optimally 30 - 150 seconds.
 7. Process according to one of Claims 1 to 5, characterised in that the mixing of the granulate with the powdery components takes place in a fast moving mixer at a speed between 250 and 3000 r.p.m. and during a sojourn time in the mixer of 1 - 300 seconds, or preferably 2 - 180 seconds, or optimally 3 - 90 seconds.
 8. Process according to one of Claims 1 to 7, characterised in that the mixture of granulate(s) and powdery components is subjected to at least four, or preferably at least eight, or optimally at least 10 mixer rotations.
 9. Process according to one of Claims 1 to 8, characterised in that a tenside compound containing at least 30 percent in weight (in relation to the tenside compound) of fatty alcohol sulphate is added as the last powdery upgrading component in the course of the mixing process and that the mixture is, after addition of the fatty alcohol sulphate compound, subjected to at least four mixer rotations during a sojourn time in the mixer of 1 - 180 seconds, or preferably 10 - 150 seconds, or optimally 20 - 120 seconds.

10. Process according to one of Claims 1 to 8, characterised in that a tenside compound containing at least 30 percent in weight (In relation to the tenside compound) of fatty alcohol sulphate as well as zeolite X are added as powdery upgrading components in the course of the mixing process, with zeolite X preferably being added as the last upgrading component, and that the mixture is, after addition of the fatty alcohol sulphate compound and zeolite X, subjected to at least four mixer rotations during a sojourn time in the mixer of 1 - 180 seconds, or preferably 10 - 150 seconds, or optimally 20 - 120 seconds.
11. Process according to one of Claims 9 or 10, characterised in that the fatty alcohol sulphate compound is added as a powdery upgrading component in such a quantity that the mixture to be compressed contains at least 2 percent in weight, or preferably at least 4 percent in weight, or optimally more than 5 percent in weight of fatty alcohol sulphate.
12. Process according to one of Claims 1 to 11, characterised in that compression shaping of the mixture takes place at a pressure of 10 - 150 N/cm², or preferably 15 - 100 N/cm² or optimally 20 - 100 N/cm² and a temperature of 10 - 80°C, or preferably 15 - 70 °C, and optimally 20 - 60°C.